

100353

Gas Chromatography/Mass Spectrometer- see EPA Method 625 for details. Analysis is performed with a DB-5 0.25mm x 30 m column, or equivalent, inserted into the source of the mass spectrometer.

6. Reagents

Organic free water- Organic free water is defined as water in which no interferences are observed at the method detection limit for each parameter of interest.

Sulfuric acid solution (1+1 vol/vol).

Acetone and Methylene chloride- Pesticide or distilled in glass quality.

Sodium Sulfate- (ACS) Granular, anhydrous. Purify by heating in a muffle furnace at 400 C overnight.

Analytical and surrogate standards - see method 625 for additional details.

7. Calibration

Instrument calibration should be with multiple point calibration curves. See method 625 or the EPA Contract Laboratory Program Statement of Work Organic Analysis for additional details.

8. Quality Control

Good quality control procedures are essential for obtaining meaningful data. All laboratories should have an effective quality control/quality assurance program in place. For additional details see method 625 or the EPA Contract Laboratory Program Statement of Work Organic Analysis .

9. Sample Collection, Preservation and Handling

Tissue samples should be obtained by personnel trained in the collection of tissue samples for trace organic analysis. This will require knowledge of biology so that the proper tissue samples are obtained and knowledge of potential contamination problems arising from trace environmental analysis.

The whole fish should be kept refrigerated until the tissue sub-samples can be obtained. The collected tissue sub-samples should be placed in precleaned glass jar with Teflon lid liners and kept frozen until time of analysis. The frozen sub-samples should be thawed just enough to obtain a representative portion and not be left at room temperature for extended periods of time. Sample holding times for tissues have not been determined. Ideally, the tissue samples should be analyzed as soon as possible after collection. The sample extracts should be kept refrigerated and analyzed within 40 days of extraction.

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10. Tissue sample preparation.

1. Weigh approximately 50 g of thawed tissue in a 250 ml centrifuge bottle. Add an appropriate surrogate or standard spiking solutions.
2. Add 100 ml of acetone to the centrifuge bottle. Homogenize the tissue with the tissue homogenizer operating at full speed for 3 minutes.
3. Decant the acetone extract into a funnel containing a Whatman 41 filter paper. Collect the filtered extract in a 500 ml Erlenmeyer flask.
4. Extract the tissue two more times by the above procedure. All acetone extracts are combined. After the final extraction transfer the tissue to the filter and rinse with additional acetone.
5. Add a boiling chip to the Erlenmeyer flask equipped with a 3 ball Snyder column. Concentrate the acetone extract to approximately 25 ml volume or until it begins to separate into two phases (one phase is water).
6. After the extract has cooled, add approximately 200-250 ml of methylene chloride followed by enough anhydrous sodium sulfate to dry the sample extract. After the extract is dry, transfer it to a Kuderna-Danish (K-D) concentration apparatus. Concentrate the extract to a volume of approximately 5 ml.
7. Load the entire extract into the sample injection loop of the GPC system. Include all rinsings. Pass the sample through the column containing Biobeads SX-3 and collecting the previously determined fraction containing compounds of interest in a K-D apparatus.

The extract loaded on the GPC column should have no more than 0.1g lipids/ ml of extract ~~is recommended~~ for best cleanup performance.

Additional details regarding set up and calibration of the GPC system can be found in the EPA Contract Laboratory Program Statement of Work for Medium and Low Level Organic Analysis.

8. Add a 3 ball Snyder column to the K-D and concentrate the extract to a volume of approximately 5 ml. Allow the extract to cool.
9. Add the extract to approximately 1 liter of organic free water, adjust the pH to less than 2 with sulfuric acid and extract the water three times with 100 ml (3 x 100ml) methylene chloride. Combining all extracts in a K-D and concentrate the extract to approximately 5 ml. Allow extract to cool.
10. Load the extract with solvent rinsings into the sample injection valve of the GPC system equipped with a Biobead SX-8 column. Inject the sample and collect the desired fraction in a K-D apparatus. Place a three ball Snyder column on the K-D and concentrate to approximately 5 ml.
11. Continue concentration of the collected eluant to a volume of 1 ml using the steam bath and nitrogen evaporation bath. The extract is now ready for analysis by GC/MS.
12. The GC/MS scanning parameters are specified in either Method 625 or the CLP SOW. The GC conditions should be optimized for separation of the compounds of interest. Sample injection should be under splitless injection conditions, with the starting column

*why acid
as acid?*

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temperature near 30 C. The injected sample volume is 2 microliters.

11. Daily GC/MS Performance Tests

See EPA Method 625 for details.

12. Gas Chromatography/ Mass Spectrometry

See EPA Method 625 for details.

13. Qualitative Identification

See EPA Method 625 for details.

14. Calculations

See EPA Contract Laboratory Program Statement of Work Organic Analysis for calculations involving solid matrices.

15. Method Performance

The following recoveries have been obtained for replicate analysis of fish tissue by this method. Cleanup method A utilizes the following sequence: GPC Cleanup with Biobeads SX-3, water back extraction, GPC cleanup with Biobeads SX-8. Cleanup method B involves using the following sequence: water back extraction, GPC Cleanup with Biobeads SX-3, GPC cleanup with Biobeads SX-8. The spiking of all priority pollutant target compounds was done at two levels, these being 40 ug/kg and 80 ug/kg.

Compounds	<u>Method A</u>	Method B
Grand Mean (60 compounds)	90(41%)a 96(35%)b	78(41%)a 87(39%)b
Chlorobenzenes (4 compounds)	46(25%)a 66(41%)b	39(29%)a 48(28%)b
Phenols (14 compounds)	111(47%)a 99(17%)b	84(45%)a 90(41%)b
PAHs (22 compounds)	97(18%)a 104(27%)b	83(20%)a 93(18%)b

(x%) is average relative standard deviation

a- spiked at 80 ug/kg

b- spiked at 40 ug/kg

16. References

1. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, U.S. Environmental Protection Agency, July 1982, EPA-600/4-82-057.

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2. U.S. Environmental Protection Agency, Contract Laboratory Program, Statement of Work, Organic Analysis, February 1988.

3. Stalling, D.L., Tindle, R.C., Johnson, J.L.; Jour. Off. Anal. Chem.; 55(1972), P.32-38.

4. Federal Register, EPA Method 625, 1984.

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Fish Spike Data

40 Mean Std. Dev. % RSD
~~80~~ ppb

Compound	Mean	Std. Dev.	% RSD
1,2,4-TRICHLOROBENZENE	74.19	6.72	9.1%
1,2-DICHLOROBENZENE	58.11	14.16	24.4%
1,3-DICHLOROBENZENE	42.09	7.70	18.3%
1,4-DICHLOROBENZENE	51.74	11.66	22.5%
2,4,5-TRICHLOROPHENOL	79.22	18.03	22.8%
2,4,6-TRICHLOROPHENOL	139.84	20.56	14.7%
2,4-DICHLOROPHENOL	90.53	4.94	5.5%
2,4-DIMETHYLPHENOL	129.41	36.63	28.3%
2,4-DINITROTOLUENE	70.09	18.36	26.2%
2,6-DINITROTOLUENE	73.00	11.92	16.3%
2-CHLORONAPHTHALENE	89.92	5.92	6.6%
2-CHLOROPHENOL	72.77	27.84	38.3%
2-METHYLNAPHTHALENE	85.47	5.72	6.7%
2-METHYLPHENOL	110.81	14.81	13.4%
2-NITROPHENOL	66.97	3.99	6.0%
4,6-DINITRO-2-METHYLPHEN	33.88		
4-BROMOPHENYL-PHENYLETHE	105.91	27.99	26.4%
4-CHLOROPHENYL-PHENYLETH	102.16	16.24	15.9%
4-CHLORO-3-METHYLPHENOL	82.31	12.95	15.7%
4-METHYLPHENOL	139.00	37.12	26.7%
ACENAPHTHENE	103.16	8.70	8.4%
ACENAPHTHYLENE	99.50	5.01	5.0%
ANTHRACENE	79.03	24.55	31.1%
BENZO(A)ANTHRACENE	114.88	12.47	10.9%
BENZO(A)PYRENE	103.00	12.47	12.1%
BENZO(B)FLUORANTHENE	106.75	7.08	6.6%
BENZO(G,H,I)PERYLENE	83.91	7.24	8.6%
BENZO(K)FLUORANTHENE	106.75	7.08	6.6%
BIS(2-CHLOROETHOXY)METHA	88.31	6.21	7.0%
BIS(2-CHLOROETHYL)ETHER	67.34	16.81	25.0%
BIS(2-CHLOROISOPROPYL)ET	68.94	11.47	16.6%
BUTYLBENZYLPHTHALATE	156.67	41.27	26.3%
CHRYSENE	89.16	13.37	15.0%
DIBENZOFURAN	94.41	8.91	9.4%
DIBENZ(A,H)ANTHRACENE	83.38	12.85	15.4%
DIETHYLPHTHALATE	113.91	20.79	17.9%
DIMETHYL PHTHALATE	109.84	9.75	8.9%
FLUORANTHENE	88.06	14.38	16.3%
FLUORENE	101.50	7.28	7.2%
HEXACHLOROBENZENE	106.91	19.85	18.6%
HEXACHLOROBUTADIENE	71.06	6.84	9.6%
HEXACHLOROETHANE	44.19	18.69	42.3%
INDENO(1,2,3-CD)PYRENE	81.47	11.43	14.0%
ISOPHORONE	147.91	61.27	41.4%
NAPHTHALENE	81.44	7.19	8.8%
NITROBENZENE	94.89	41.15	43.4%
N-NITROSO-DI-N-PROPYLAMI	70.34	9.66	13.7%
PENTACHLOROPHENOL	214.13	64.40	30.1%
PHENANTHRENE	140.50	21.85	15.6%
PHENOL	89.50	24.06	26.9%
PYRENE	111.88	13.78	12.3%
Ave	94.35	17.02	17.5%
Min	33.88	3.99	5.0%
Max	214.13	64.40	43.4%

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Fish Spike Data

Compound	Mean	Std. Dev.	% RSD
	ppb		
1,2,4-TRICHLOROBENZENE	61.56	13.09	21.3%
1,2-DICHLOROBENZENE	41.13	14.96	36.4%
1,3-DICHLOROBENZENE	31.85	17.26	54.2%
1,4-DICHLOROBENZENE	35.58	16.26	45.7%
2,4,5-TRICHLOROPHENOL	108.56	33.73	31.1%
2,4,6-TRICHLOROPHENOL	126.00	7.50	6.0%
2,4-DICHLOROPHENOL	86.63	7.35	8.5%
2,4-DIMETHYLPHENOL	104.06	22.11	21.2%
2,4-DINITROTOLUENE	65.38	22.42	34.3%
2,6-DINITROTOLUENE	75.25	10.37	13.8%
2-CHLORONAPHTHALENE	79.56	6.03	7.6%
2-CHLOROPHENOL	71.50	11.88	16.6%
2-METHYLNAPHTHALENE	80.63	9.96	12.4%
2-METHYLPHENOL	111.88	35.13	31.4%
2-NITROPHENOL	56.88	7.17	12.6%
4,6-DINITRO-2-METHYLPHEN	37.30	14.95	40.1%
4-BROMOPHENYL-PHENYLETHE	80.69	10.97	13.6%
4-CHLOROPHENYL-PHENYLETH	90.44	4.80	5.3%
4-CHLORO-3-METHYLPHENOL	126.19	66.19	52.5%
4-METHYLPHENOL	117.25	37.74	32.2%
ACENAPHTHENE	88.88	4.32	4.9%
ACENAPHTHYLENE	92.06	2.48	2.7%
ANTHRACENE	84.49	40.05	47.4%
BENZO(A)ANTHRACENE	110.44	10.46	9.5%
BENZO(A)PYRENE	92.75	4.61	5.0%
BENZO(B)FLUORANTHENE	93.19	7.30	7.8%
BENZO(G,H,I)PERYLENE	90.94	20.33	22.4%
BENZO(K)FLUORANTHENE	93.92	8.44	9.0%
BIS(2-CHLOROETHOXY)METHA	74.13	7.10	9.6%
BIS(2-CHLOROETHYL)ETHER	51.56	9.01	17.5%
BIS(2-CHLOROISOPROPYL)ET	48.18	15.99	33.2%
BUTYLBENZYLPHTHALATE	109.42	21.64	19.8%
CHRYSENE	88.25	14.99	17.0%
DIBENZOFURAN	88.58	3.40	3.8%
DIBENZ(A,H)ANTHRACENE	98.63	19.99	20.3%
DIETHYLPHTHALATE	105.88	9.83	9.3%
DIMETHYL PHTHALATE	101.94	5.39	5.3%
FLUORANTHENE	101.75	35.08	34.5%
FLUORENE	95.88	6.11	6.4%
HEXACHLOROBENZENE	96.44	11.10	11.5%
HEXACHLOROBUTADIENE	54.98	15.80	28.7%
HEXACHLOROETHANE	30.25	14.32	47.3%
INDENO(1,2,3-CD)PYRENE	93.44	19.14	20.5%
ISOPHORONE	71.94	6.55	9.1%
NAPHTHALENE	69.69	12.61	18.1%
NITROBENZENE	89.25	15.10	16.9%
N-NITROSO-DI-N-PROPYLAMI	61.56	5.58	9.1%
PENTACHLOROPHENOL	172.50	27.00	15.7%
PHENANTHRENE	88.19	27.61	31.3%
PHENOL	76.31	23.49	30.8%
PYRENE	94.06	18.59	19.8%
Ave	84.27	15.95	20.4%
Min	30.25	2.48	2.7%
Max	172.50	66.19	54.2%

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Fish Spike Data

Compound	Mean	Std. Dev.	% RSD
	400-800ppb		
1,2,4-TRICHLORO BENZENE	63.75	4.13	6.5%
1,2-DICHLORO BENZENE	36.00	2.35	6.5%
1,3-DICHLORO BENZENE	24.25	2.17	8.9%
1,4-DICHLORO BENZENE	27.75	2.28	8.2%
2,4,5-TRICHLOROPHENOL	88.75	21.28	24.0%
2,4,6-TRICHLOROPHENOL	100.00	15.67	15.7%
2,4-DICHLOROPHENOL	82.50	7.89	9.6%
2,4-DIMETHYLPHENOL	60.00	35.80	59.7%
2,4-DINITROTOLUENE	76.75	5.54	7.2%
2,6-DINITROTOLUENE	80.50	9.86	12.3%
2-CHLORONAPHTHALENE	81.25	8.70	10.7%
2-CHLOROPHENOL	73.50	6.54	8.9%
2-METHYLNAPHTHALENE	81.50	7.92	9.7%
2-METHYLPHENOL	78.75	19.56	24.8%
2-NITROPHENOL	66.00	7.55	11.4%
4,6-DINITRO-2-METHYLPHEN	62.25	11.71	18.8%
4-BROMOPHENYL-PHENYLETHE	85.50	5.94	6.9%
4-CHLOROPHENYL-PHENYLETH	91.00	5.79	6.4%
4-CHLORO-3-METHYLPHENOL	96.50	12.34	12.8%
4-METHYLPHENOL	95.25	6.53	6.9%
ACENAPHTHENE	82.25	8.50	10.3%
ACENAPHTHYLENE	90.25	14.10	15.6%
ANTHRACENE	72.25	22.25	30.8%
BENZO(A)ANTHRACENE	103.25	5.63	5.5%
BENZO(A)PYRENE	86.75	10.16	11.7%
BENZO(B)FLUORANTHENE	92.75	13.37	14.4%
BENZO(G,H,I)PERYLENE	72.75	16.84	23.2%
BENZO(K)FLUORANTHENE	92.50	12.97	14.0%
BIS(2-CHLOROETHOXY)METHA	67.75	9.91	14.6%
BIS(2-CHLOROETHYL)ETHER	45.75	4.47	9.8%
BIS(2-CHLOROISOPROPYL)ET	48.75	10.03	20.6%
BUTYLBENZYLPHTHALATE	92.25	42.63	46.2%
CHRYSENE	90.75	13.55	14.9%
DIBENZOFURAN	86.75	7.66	8.8%
DIBENZ(A,H)ANTHRACENE	77.75	13.05	16.8%
DIETHYLPHTHALATE	94.25	6.22	6.6%
DIMETHYL PHTHALATE	91.50	1.50	1.6%
FLUORANTHENE	92.75	8.64	9.3%
FLUORENE	91.00	9.25	10.2%
HEXACHLORO BENZENE	92.25	10.03	10.9%
HEXACHLOROBUTADIENE	51.00	5.79	11.3%
HEXACHLOROETHANE	22.50	2.50	11.1%
INDENO(1,2,3-CD)PYRENE	78.25	12.97	16.6%
ISOPHORONE	65.25	10.57	16.2%
NAPHTHALENE	66.50	6.87	10.3%
NITROBENZENE	55.25	7.53	13.6%
N-NITROSO-DI-N-PROPYLAMI	58.75	6.76	11.5%
PENTACHLOROPHENOL	160.00	37.42	23.4%
PHENANTHRENE	93.50	5.72	6.1%
PHENOL	71.00	12.14	17.1%
PYRENE	93.00	13.36	14.4%
Ave	77.08	11.02	14.2%
Min	22.50	1.50	1.6%
Max	160.00	42.63	59.7%

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U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal

☐ Telephone Request

- A. EPA Region and Client: EPA Region III
- B. Regional Representative: Colleen K. Walling
- C. Telephone Number: (301) 266-9180
- D. Date of Request:
- E. Site Name: Standard Chlorine Delaware City, De.

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:

Digestion And Analysis of 2 low concentration whole body fish samples for TAL metals by the 7/88 CLP-SOW with revisions. Fish are to be composited and homogenized by the laboratory.

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):

2 low concentration whole body fish samples for TAL metals only by the CLP-SOW (7/88) with revisions.

The awarded laboratory is responsible for meeting all requirements as specified in this client request. Any changes in method(s) or other specifications must be approved by Region III prior to the award. The referenced Statement of Work must be used including all current revisions of that SOW. If these stipulations are not met, Region III will recommend review for reduced payment.

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3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund Enforcement OTGB03 NPHC

SAS Approved By:

4. Estimated date(s) of collection:

5. Estimated date(s) and method of shipment:

6. Approximate number of days results required after lab receipt of samples:

DATA PACKAGE due 35 days AFTER VTSD of last sample

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

AS per CLP-SOW (7/88) with revisions

~~AS~~

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

Determine (%) Solids AS per (7/88) CLP-SOW

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, blank results, duplicate results, chain of custody forms, SAS request forms, SAS packing list(s) or traffic report(s), copy of airbill(s), and copies of analyst's logbooks (signed by analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labeled with the complete EPA sample number as it appears on chain of custody and CLP paperwork.

The case narrative must document all problems encountered and the subsequent resolutions. List instrumentation and methods employed for analysis.

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11. Name of sampling/shipping contact:

Phone:

12. Data Requirements

Parameter

Detection Limit

Precision Desired
(+ or - Concentration)

AS per CLP-SOW (7/88 with revisions)

13. QC Requirements

Audits Required

Frequency of Audits

Limits
(Percent or Concentration)

AS per CLP-SOW (7/88 with revisions)

14. Action Required if Limits are Exceeded

AS per CLP-SOW (7/88 with revisions)

15. Request prepared by:

C. Sands

Date:

1/27/90

16. Request reviewed by:

Date:

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

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